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**(54) ULTRAVIOLET-CURABLE CATIONIC ELECTRODEPOSITION COATING COMPOSITION
SUITABLE FOR PLATED MATERIAL**

(57)Abstract:

PURPOSE: To obtain an electrodeposition coating composition which is applicable to not only ordinary metals but also plated plastic or die-cast-metal articles, is rich in adhesion and flexibility, and imparts excellent anticorrosion by incorporating a polyfunctional acrylate and a specific resin capable of undergoing cationic electrodeposition as the active ingredients.

CONSTITUTION: This composition contains, as the active ingredients, 10-70, excluding 70, pts.wt. polyfunctional acrylate having three or more acryloyl groups per molecule and 30-90, excluding 90, pts.wt. resin which is capable of being cationically electrodeposited and has an average mol.wt. of 2,000-30,000. The resin is a copolymer of 1-20, excluding 20, pts.wt. vinyl monomer having a tertiary amino group and 80-99, excluding 99, pts.wt. at least one member selected from among hydroxyesters of α,β -unsaturated ethylenic monocarboxylic acids, alkyl esters of α,β -unsaturated ethylenic monocarboxylic acids, and α,β -unsaturated ethylenic compounds.

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CLAIMS

[Claim(s)]

[Claim 1] The ultraviolet curing mold cation electrodeposition paint constituent suitable for the plating material characterized by containing under 90 weight sections for the resin of the average molecular weight 2,000-30,000 which has cation electrodeposition nature for the polyfunctional acrylate which has three or more acryloyl radicals in a molecule under 70 weight sections more than 10 weight sections as an active principle more than 30 weight sections.

[Claim 2] The resin which has said cation electrodeposition nature following (a) more than 1 weight section Under 20 weight sections Or (b-2) (b-3) it is a copolymerization object more than the under 99 weight sections 80 weight section about one sort or two sorts or more of mixture the following (b-1) — The ultraviolet curing mold cation electrodeposition paint constituent suitable for the plating material according to claim 1 characterized by for the average molecular weight being 3,000-30,000, and being resin which has the 3rd class amino group in the side chain of the copolymerization object.

(a) The 3rd class amino-group content vinyl monomer (b-1) alpha, beta-ethylene nature partial saturation monocarboxylic acid hydroxy ester (b-2) alpha, beta-ethylene nature partial saturation monocarboxylic acid alkyl ester (b-3) alpha, beta-ethylene nature unsaturated compound [claim 3] The resin which has said cation electrodeposition nature is [Formula 1] in a polymer.

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R

However, R is an ultraviolet curing mold cation electrodeposition paint constituent suitable for the plating material according to claim 1 characterized by being polyurethane of the average molecular weight 2,000-10,000 containing the 3rd class amino group shown by the with a carbon number of four or less alkyl group.

[Claim 4] Said electrodeposition paint constituent is an ultraviolet curing mold cation electrodeposition paint constituent suitable for claim 1 characterized by including a coating, pigments, or those mixture below 20 weight sections thru/or a plating material given in three if needed.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to the ultraviolet curing mold cation electrodeposition paint constituent which gives anti-corrosiveness, such as a water resisting property, chemical resistance, artificial-proof ****, and fingerprint-proof nature, and a variegated color variation to a plating material.

[0002]

[Description of the Prior Art] Electropainting is a painting method which is immersed, is made to undergo a conductive coated object in the quarry of film former which water was distributed and gave the charge so that it might be well-known, and performs printing processing after electrica coagulation with reference for example, J.D. Col. Chem. Assoc. 63, 492 (1980).

[0003] As main descriptions of this painting method, there being few losses of a coating and automatic management of paint are easy, and film formation is possible to homogeneity to that reduction of labor costs can be aimed at, that variegated coated objects can be processed to coincidence, and the internal side and the edge section of a coated object, and a coating is attached, and it is mentioned that a surroundings property is good etc. Moreover, the object for prizes of the electropainting which uses a drainage system coating also from the field of environmental pollution and disaster prevention is carried out.

[0004] Electropainting is divided roughly into anion electropainting and cation electropainting.

Since a coated object is used as anode, there being little studion of a substrate metal or a chemical film and paint film show badly, therefore paint film resin itself acts as a

corrosion retarder (corrosion retarder), and since advanced corrosion resistance is demonstrated, cation electropainting is broadly used as a painting method of an automobile car body and the Industrial Supplies Department gate.

[0005]

[Problem(s) to be Solved by the Invention] However, since the paint film used with said conventional technique is thermosetting, curing temperature has the problem that it is as high as 100 degrees C or more, and cannot be used for heat to materials, such as work plastics.

[0006] Although there is the approach of using paint for ultraviolet curing and stiffening by UV irradiation in order to solve this, it consists of ultraviolet-rays hardenable oligomer, a monomer, a photopolymerization initiator, a crosslinker, etc., and is called the high speed and the non-solvent hard. Since other constituents are diluted with a monomer instead of an organic solvent, there is a problem of the bad influence to the body by monomer scattering at the time of paint. Moreover, the paint film obtained from this type of coating has the problem that it is weak and adhesion with a base material is also bad, although the thing of a high degree of hardness is obtained. On a smooth base material, this point is a problem especially like a plating coat.

[0007] The purpose of this invention is offering the electrodeposition paint constituent which can apply not only to a common metal but to the plating product made from plastics or glass casting, and is rich in adhesion and flexibility, and gives the outstanding anti-corrosiveness, and transparency, gloss and a variegated color variation.

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2005/12/06

JP.DU-268025A [DETAILED DESCRIPTION]

3/8 ← →

sort or two sorts or more of mixture, and the average molecule is 3,000-30,000, and or (b-2) (b-3) makes it indispensable to have the 3rd class amino group in the side chain of the copolymerization object.

[0017] (a) The 3rd class amino-group content vinyl monomer, for example, (meta), acrylic-acid dimethyl dimethylaminomethyl, acrylic-acid (meta), dimethylaminomethyl (Meta) alpha-(b-1), such as vinylpyridine, beta-ethylene nature partial saturation monocarboxylic acid hydroxy ester. For example, (meta), acrylic-acid hydroxyethyl, acrylic-acid (Meta) hydroxymethyl, alpha-(b-2), such as acrylic-acid hydroxy butyl, beta-ethylene nature partial saturation monocarboxylic acid alkyl ester. (Meta) For example, (meta), a methyl acrylate, an ethyl acrylate (meta), acrylic-acid (meta) propyl, Butyl acrylate, acrylic-acid (meta)-2-ethyl hexyl, (Meta) Acrylon-100, acrylic-acid (meta) BORNIRU, acrylic-acid (meta) butyl, (Meta) alpha-(b-3), such as acrylic-acid cyclohexyl and acrylic-acid (meta) phenyl ethyl, beta-ethylene nature unsaturated compound. (Meta) For example, the 3rd class (b) amino-group content vinyl monomers, such as styrene, methyl styrene and vinylcarbazole what it is the indispensable component of a graft, etc. give the cation electrodeposition nature and the monomer of a parentheses give the grafted of a paint film and a substrate plating coat — it is — that polymerization rate — 1 — 20 weight section — is 2 — 10 weight section preferably. If there are too few polymerization rates, moisture powder is not carried out, and electrodeposition nature shows abnormality electrodeposition low and cannot form a uniform paint film. Also when many [too], the conductivity of a coating goes up, and it becomes the cause which causes abnormality electrodeposition too.

[0018] (b) With copolymerization with the 3rd class amino-group content vinyl monomer of this (b-1) and beta-ethylene nature partial saturation monocarboxylic acid hydroxy ester is used in order to give adhesion with the auxiliary component of the formation of monomer powder, and a substrate plating coat. Although loadings can be chosen in the large range, it is 3 — 20 weight section preferably. If many [too], since the water absorption of a paint film will increase, it is not desirable.

[0019] alpha-(b-2) and beta-ethylene nature partial saturation monocarboxylic acid alkyl ester is used in the making the flexibility of a paint film, flow nature, etc. give purpose, and loadings can be chosen in the large range if needed.

[0020] alpha-(b-3) and beta-ethylene nature unsaturated compound are used if needed as a modifier of resistance of improvement in physical properties, such as the water resisting property of a paint film, and chemical resistance, and 1% of a paint film. When many [too], in order to increase the brittleness of a paint film, 30 weight sections are limits.

[0021] A well-known radical polymerization method is applied as the copolymerization approach. That is, it is obtained by the cation polymerization using a suitable solvent, for example, a methanol, ethanol, isopropanol alcohol, methyl cellosolve, allylcellosolve, butyl cellosolve, butyl cellosolve acetate, toluene, a xylene, etc.

[0022] As a polymerization initiator, 2 and 2-azobis (2,4-dimethylvaleronitrile), a benzoyl peroxide, etc. are mentioned.

[0023] The obtained copolymer neutralizes the basicity and gives water-dispersion [required for electrodeposition]. As an acid required for neutralization, inorganic acids, such as organic acids, such as an oxalic acid, formic acid, a propionic acid, and a lactic acid, or a sulfuric acid, and phosphoric acid, are mentioned.

[0024] As the resin which has cation electrodeposition nature — the above (a) and (b-1) — in or (b-2) (b-3), when a copolymerization object with one sort or two sorts or more of mixture to used, the 3rd class amino group and hydroxyl group which were added to the side chain give adhesion with a substrate plating coat. Moreover, although contained as a copolymerization component (b-3) (b-2), the flexibility of a paint film and a degree of hardness can be adjusted if needed by choosing a suitable thing from inside.

[0025] Moreover, the resin which has cation electrodeposition nature is [0026].

[Formula 3]

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JP.DU-268025A [DETAILED DESCRIPTION]

[0009]

[Means for Solving the Problem] This invention is an ultraviolet curing mold cation electrodeposition paint constituent suitable for the plating material characterized by containing under 30 weight sections for the resin of the average molecular weight 2,000-30,000 which has cation electrodeposition nature for the polycationic acrylate which has three or more acryloyl radicals in a molecule under 70 weight sections more than 10 weight sections as an active principle more than 30 weight sections.

[0010] Moreover, the resin with which this invention has said cation electrodeposition nature — the following — (a) — more than 1 weight section — under 20 weight sections and the following (b-1) — it is a copolymerization object more than the under 99 weight sections 80 weight section about one sort or two sorts or more of mixture, and the average molecular weight is 3,000-30,000, and or (b-2) (b-3) is characterized by being resin which has the 3rd class amino group in the side chain of the copolymerization object.

[0011] (a) For the 3rd class amino-group content vinyl monomer (b-1) alpha, beta-ethylene nature partial saturation monocarboxylic acid hydroxy ester (b-2) alpha, beta-ethylene nature unsaturated compound, and this invention, the resin which has said cation electrodeposition nature is [0011] in a polymer.

[Formula 2]

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[0012] However, R is characterized by being polyethers of the average molecular weight 2,000-10,000 containing the 3rd class amino group shown by the with a carbon number of four or less alkyl group.

[0013] Moreover, this invention is characterized by said electrodeposition paint constituent containing a coating, pigments, or those mixture below 20 weight sections if needed.

[0014]

[Function] If this invention is followed, it will consider as the active principle which has 10 — 70 % of the weight of acrylate which has three or more acryloyl radicals which the radical polymerization reaction by ultraviolet rays produces, and 90 — 30 % of the weight of resin which has cation electrodeposition nature. When the resin which has cation electrodeposition nature is electrodeposited by the coated object, acrylate resin, a paint film is formed, and by ultraviolet rays, acrylate open up a radical polymerization and hardens. Since an acryloyl radical is in [three or more] 1 molecule at this time, the paint film produced at a polymerization reaction serves as the three-dimensional network structure, and the paint film which has preduced strength for a short time is obtained. Since hardening processing can perform using ultraviolet rays as a method of hardening these paint films, without raising the temperature of a coated object, it can apply to the plasted plating product made from ABS plastic etc. and there is no possibility that heating etc. may arise by elevated-temperature baking finish also to the plating products made from die casting. Since it can furthermore be characterized compared with the common electrodeposition paint in which the setting time needs elevated-temperature baking, improvement in working efficiency can also be aimed. It becomes insufficient [20 or less % of the weight] electrodepositing the resin with which the polycationic acrylate which has three or more acryloyl radicals becomes inadequate [carrying out a radical polymerization and hardening at 10 or less % of the weight], and has cation electrodeposition nature.

[0015] As polyfunctional acrylate which has said three or more acryloyl radicals, there are trimethylpropane triacrylate, pentaerythritol three, hexafunctional triacrylate, triacryl methyl TAKU, etc. etc. Preferably, compatibility with the resin which has cation electrodeposition nature is good, and the polyether acrylates which denaturalized in Urethane or more organic functions, many organic functions from which the more excellent paint film engine performances obtained, urethane acrylates, epoxy acrylates, etc. are good.

[0016] As the resin which has cation electrodeposition nature — the following — 1 — 20 weight section of (a), and (b-1) — it is a copolymerization object with 80 — 99 weight section of one

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2005/12/06

JP.DU-268025A [DETAILED DESCRIPTION]

4/8 ← →

[0027] However, R may be polyurethane of the molecular weight 2,000-10,000 containing the 3rd class amino group shown by the with a carbon number of four or less alkyl group. Generally, although polyurethane is compounded by the reaction of diisocyanate and diol, diol and diisocyanate (diisocyanate), in order to introduce said the 3rd class amino group, it becomes an indispensable condition to blend the 3rd class amino diol with some diisocyanate alcohol.

[0028] The 3rd class amino diol of (b-1) has, for example, N-methyl diethanolamine, N-ethyl diethanolamine, N-propyl diethanolamine, N-butyl diethanolamine, etc. The diol of either of (b-2) has ethylene glycol, 1,3-propylene glycol, 1,4-butanediol, 1,6-hexamethylene diol, a para xylylene glycol, polyisobutylene diol used in the above, and other diols. 1,3 is desirable at a mole ratio. If there are four the 3rd class amino diol components that this electropainting will become impossible, and abnormalities will be caused to a paint film appearance if many [again / too].

[0029] Moreover, the diisocyanate of being another component of polyurethane composition (a-2) has 4 and 4-diphenylmethane diisocyanate, tolylene diisocyanate, hexamethylene diisocyanate, isophorone diisocyanate, 4,4-dimethylbenzyl diisocyanate (isocyanate), trimethylhexamethylene diisocyanate, 1,1,1-trimethylolpropane diisocyanate, etc.

[0030] The addition polymerization of the diols shown above and the diisocyanate is carried out with a conventional method in the solvent which does not have active hydrogen, such as benzene, toluene, xylene, an acetone, a methyl ethyl ketone, methyl acetate, and ethyl acetate.

[0031] The obtained polymer neutralizes the basicity and gives water-dispersion [required for electrodeposition]. As an acid required for neutralization, inorganic acids, such as organic acids, such as an acetic acid, formic acid, a propionic acid, and a lactic acid, and phosphoric acid, are mentioned.

[0032] When the polyurethane containing said the 3rd class amino group is used as resin which has cation electrodeposition nature, an adhesive property, a water sealing property, and the paint film excellent in especially flexibility can be obtained.

[0033] The class product described above is prepared by the electrodeposition paint by the following (1) or the approach of (2).

[0034] (1) Mix with one sort or two sorts or more of copolymer among hydroxy ester [of the neutralized 3rd class amino-group content vinyl monomer and alpha and beta-ethylene nature partial saturation monocarboxylic acid] or alpha, alkyl ester [of beta-ethylene nature partial saturation monocarboxylic acid] or alpha, and beta-ethylene nature unsaturated compounds, diol polyfunctional acrylate with ion exchange water, and make it into 8 — 15% of pitches.

[0035] (2) Mix with the polyurethane containing said the 3rd class amino group neutralized at polyfunctional acrylate, diol with ion exchange water, and make it 8 — 15% of pitches.

[0036] Moreover, in order to give a photopolymerization initiator or a color variation to the ultraviolet curing mold cation electrodeposition paint adjusted by (1) or (2) if needed, a color or pigment may be added. As a photopolymerization initiator used, there are 4-dimethylaminobenzene, 4-dimethylaminobenzyl benzene, 2-dimethylaminobenzyl ketone, a benzophenone, BENZO 161, 2-hydroxy-2-methylpropiophenone, etc. for example. Moreover, as a color used, monobutyl dye is desirable. As a pigment, they are metallic oxides, such as a share of an organic pigment, for example, an iron system, a phthalocyanine system, a metallic complex system, and the Quinacridone system or an inorganic pigment, for example, a titanium dioxide, and ferric oxide, a barium sulfate, etc. In addition, there is well-known carbon black.

[0037] Although what is necessary is just to choose the mixed rate of a color or a pigment by the share of the color made into the purpose, it must not exceed 20% of the weight of the whole ultraviolet curing mold cation electrodeposition paint. If this is exceeded, the aforementioned description of this invention will be lost.

[0038]

[Example] Hereafter, although it explains more concretely that this invention is also an

example, this invention is not limited to this.

[0039] Example 1 (1-A) agitator, a condenser, and a tap funnel in the 4 opening flask with which each opening was equipped Tohma (Tohmyaku) 200g of hexamethylene di-isocyanate, Dibutyl tin dilaurate 0.46g which has a catalyst in acrylic-acid-2-hydroxyethyl 116g while heating and agitating acetone 135g. The mixed liquor which added METOKINON 0.1g as a polymerization agent was dropped a uniform velocity for 10 minutes from the tap funnel, churning was continued for 30 minutes, holding at 40 more degrees C or less, and the acrylic solution made into the purpose was obtained. In addition, when absorption of 2270cm⁻¹ disappeared with the infrared absorption spectrum, it checked that the isocyanate radical had reacted completely.

[0040] (2) dimethylaminocetyl methacrylate 40g, methacrylic-acid (b-1) 2-hydroxyethyl 100g, and acrylic-acid (b-2-2- ω -butenyl 90g, (1-3) 50g of α -butyl methacrylate, 145g of methyl methacrylates, and acetone 75g are added to isopropyl alcohol 500g as a solvent. Further as a polymerization initiator The mixed liquor which added 10g for 2 and 2- ω -butyl isobutyl nitrox 1 is taught to the 4 opening flask which equipped each opening with the agitator etc. etc. (1-A). The temperature up was carried out agitating, the takes doses of the mixed liquor of the still more nearly same presentation were dropped at homogenously from the tap funnel in 90 minutes after flux initiation. It held at 85 degrees C for further 4 hours, churning was continued, and the rock solution which has the called electrodeposition nature which is these copolymer was obtained. It checked by GPC that the average molecular weight of the copolymer was 26,000.

[0041] (3-C) After adding 2-hydroxy-2-methylpropionophenone 1g as 20.4g of acrylic solutions obtained while adding 0.5g of lactic acid to 9g of obtained copolymer solutions, neutralizing and agitating (1-A), and a photopolymerization initiator, in addition, the ultraviolet curing mold cation electrodeposition paint constituent of this invention was obtained, having used the whole quantity as 1L agitating ion exchange water (1-B).

[0042] OPP-Rosin T-1 (trade name, product made from Okemura CII 101) 20g which is commercial acrylics of three or more organic functions while adding 2.7g of lactic acids to 127g of copolymer solutions obtained in the example 2 (2-C) (1-B), neutralizing and agitating. After adding 2-hydroxy-2-methylpropionophenone 1g as a photopolymerization initiator, in addition, the ultraviolet curing mold cation electrodeposition paint constituent of this invention was obtained, having used the whole quantity as 1L agitating ion exchange water.

[0043] The liquid which dissolved isocyanate diisocyanate 106.7g contained in an example 3 (2-E) (c-2) in acetone 75.8g is taught to the 4 opening flask which equipped each opening with the agitator etc. etc. (1-A). The solution which dissolved PLACCEL208 (trade name, Daikai Chemical Industries, Ltd. make) 220.7g contained in (c-2) of methacrylic and N-methylmethacrylates 24.4g contained in (c-1) in acetone 75g was dropped in 20 minutes from the tap funnel, agitating.

Temperature was held at 40 degrees C or less, churning was continued to the pan for 120 minutes, and the polyurethane solution which has the 3rd class amino group was obtained. In addition, when absorption of 2270cm⁻¹ disappeared from the infrared absorption spectrum, it checked that the isocyanate radical had reacted completely.

[0044] (3-C) After adding 2-hydroxy-2-methylpropionophenone 1g as OPP-Rosin T-16D which is commercial acrylics, and a photopolymerization initiator, having added and neutralized 2.2g of lactic acids, and agitating 71.4g of polyurethane solutions containing the obtained 3rd class amino group, in addition, the ultraviolet curing mold cation electrodeposition paint constituent of this invention was obtained, having used the whole quantity as 1L agitating ion exchange water (3-B).

[0045] After it carried out electropainting of the ultraviolet curing mold cation electrodeposition paint constituent obtained in said example 1-3 to the test piece (5mm×5mm) which performed nickel plating to ABS plastics by 10 micrometers of thickness by the approach used conventionally and 80 degrees C dried for 10 minutes, ultraviolet rays were irradiated for 2 minutes in the distance of 20cm with the UV oven (60W high pressure mercury vapor lamp) by the eye graphic company, [0046] which shows the result of an evaluation trial of the completed paint film in Table 1 with the result of the example of a comparison by this

[Table 1]

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試験項目	実施例1	実施例2	実施例3	比較例
外観	○	○	○	△
密着性	100/100	100/100	100/100	0/100
硬度	2H	3H	2H	4H
180°折曲テスト	△	○	○	×
CASS72hrs	○	○	○	○
アセトニルズ	50~50	200+	200+	200+
耐溶剤性 5hrs	○	○	○	C
5%NaOH 4.8hrs	部分白化	○	○	○
5%硫酸4.8hrs	○	○	○	○

[0047] The test method is as follows.

[0048] Besides it uses, it is based on viewing. Irregularity was accepted only for the example of a comparison for a while.

[0049] Adhesion Cross cut adhesion test JIS K 5651 in Whenever Mitsubishi Pencil Uni 160 degree folding test of 1kg loads 180 degrees of test pieces are bent. A cellulose tape is made to adhere to a fold. A peal test. O mark exfoliation nothing, x mark complete avulsion, △ mark — partial — exfoliation CASS72hrs JIS K 8817 Following O mark Normal thing acetone Rubs A 1kg load is applied to the cloth immersed in the acetone. A test piece top is made to rework. Count of round trip breakage 5hrs until a base is exposed To hot water 90 degrees C or more, the appearance after 5-hour immersion It is judgment 5%NaOH48hrs by viewing. It is visual judgment 5% sulfuric acid 48hrs about the appearance after 48-hour immersion at 25 degrees C to 5%NaOH water solution. In the urethane urethane solution which acquired the appearance after 48-hour immersion visually at 25 degrees C in the sulfuric acid water solution 5% at (1-A) of the example example 1 of a judgment comparison In addition, the ultraviolet curing mold coating constituent was obtained, having added the xylene further and having used the whole quantity of 1L, agitating 2-hydroxy-2-methylpropionophenone 1g as a photopolymerization initiator. By being immersed twice in the same test piece as having used this in the example for 5 seconds, semi-film formation was performed and desiccation and an exposure were performed on these conditions as examples 1-3. The same evaluation trial as an example was performed to this.

[0050]

[Effect of the Invention] It hardens by irradiating ultraviolet rays in ordinary temperature according to this invention as mentioned above, and a material and adhesion are good and the paint film suitable for the plating product has the anti-corrosiveness and transparency which were rich and excellent in flexibility, and made from good plastics and the dia casting of an appearance can be obtained.

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